## TECHNICAL REPORT

# Measurement of soil respiration using closed chamber method: An IRGA technique

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A closed chamber method (CC-method) using an infra-red gas analyzer (IRGA) for measuring soil respiration was examined. Two major factors which potentially cause errors: (i) volume of air sampled from the chamber; and (ii) measuring period of time, were examined in laboratory experiments. Field measurements were also conducted with both the CC-method and the open-flow IRGA method (OF-method) throughout a year. The results of laboratory experiments showed that (i) sampling volume of air should be less than 0.2% of the volume of the chamber; and (ii) the air within the chamber should be sampled several times within 20 min. Field measurements showed that soil respiration rates measured by the CC-method were not significantly different from those by the OF-method. The results of this study indicate that the CC-method is as effective for the measurement of the soil respiration rates as the OF-method.

Key words: closed chamber method; IRGA; soil respiration.

## INTRODUCTION

Methods for measuring soil respiration can be classified into three types. The first is the alkali absorption method (AA-method). Carbon dioxide that evolves from soil in a closed chamber is absorbed in a caustic solution (Witkamp 1966; Kirita 1971; Edwards & Ross-Todd 1983). The second is the closed chamber method (CC-method) where CO<sub>2</sub> in a closed chamber is sampled periodically and the efflux is computed from the concentration increase in the chamber (Matthias *et al.* 1980; Hutchinson & Mosier 1981; Rolston 1986; Mariko *et al.* 1994). Lastly, the open flow infra-red gas analyzer method (OF-method) whereby ambient air flows through a chamber, and CO<sub>2</sub> flux is calculated

from the concentration difference between inlet- and outlet-air (Witkamp & Frank 1969; Garret & Cox 1973; Nakadai et al. 1993). The AA-method has been adopted in much research for its convenience and capability to obtain many measurement plots (Kucera & Kirkham 1971; Nakane 1975; Buyanovsky et al. 1986; Singh et al. 1988). However, it has been suggested that the AA-method may underestimate or overestimate actual soil respiration rates through suppressing CO2 diffusion (Kucera & Kirkham 1971; Freijer & Bouten 1991) or through acceleration of the respiration rates under low CO<sub>2</sub> concentration in chamber (Koizumi et al. 1991; Nakadai et al. 1993). For the alternate, the OFmethod has been recently used with popularization of infra-red gas analyzer (IRGA). The OF-method is, however, less attractive for field measurements because this method requires expensive equipment and electric power supply. On the other hand, the CC-method is suitable for in situ measurements, as it is simple, rapid and capable of obtaining many measurement plots. However, the CC-method was originally designated to measure N2O emission rates from soils (Matthias et al. 1980; Hutchinson &

Received 22 December 1994. Accepted 18 May 1995.

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Mosier 1981). The CC-method using IRGA for measuring CO<sub>2</sub> emission rates from soils have not yet been examined systematically, though there have been some reports using gas chromatography (Rolston 1986; Freijer & Bouten 1991). The advantages for using IRGA are that the price of equipment and the operating costs are low, and the equipment has recently become popular in ecological laboratories, as compared with gas chromatography. The objective of this study is to establish the closed chamber technique using IRGA for measurement of soil respiration rates. In particular, the measurement system, volume of sampled air from the chamber and measuring period of time were examined.

### **MATERIALS AND METHODS**

## Measurement system

A cylindrical chamber of vinyl chloride, 21 cm in diameter and 14 cm in height, is carefully driven 4 cm into the soil more than 1 day before the measurement (Fig. 1a). The chamber is closed with a lid which has a small fan and a rubber stopper. Air in the chamber is sampled several times at certain time intervals with a micro-syringe through the stopper. A blood collection needle (Terumo, Venoject, Multiple type, Tokyo, Japan) and a vacuum vial can be used instead of the syringe. The fan should be operated for 30s just before the air sampling in order to homogenize CO<sub>2</sub> concentration in the chamber. The sampled air is transported to the laboratory, and CO<sub>2</sub> concentration of the air is analyzed by the following system (Fig. 1a). The sampled air is inserted into a gas-line in which CO<sub>2</sub> free air streamed at 0.5 l min<sup>-1</sup>, and sent to the infra-red gas analyzer (IRGA; Fuji Electric, Model ZRC, Tokyo, Japan) via a perma-pure drier (Perma Pure Product Inc., Model ZBJ). The CO<sub>2</sub> concentration in the air is determined by reading the height of the pulse monitored on a chart recorder (Graphtec, Servocorder, SR6312, Tokyo, Japan) with the calibration curve of pulse height versus CO<sub>2</sub> concentration. The increase in the CO<sub>2</sub> concentration within the chamber is expressed as a function of time (Fig. 1b). Soil respiration rate  $V_{SR}$  (mg CO<sub>2</sub> m<sup>-2</sup>  $h^{-1}$ ) is calculated by the following equation.

$$V_{SR} = 60 \times 10^{-6} \, a \, \rho \, V/S \tag{1}$$

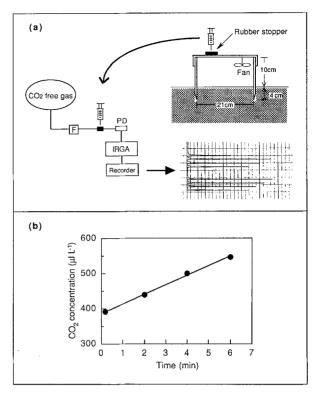


Fig. 1. (a) Soil respiration chamber for the closed chamber method and the system for measuring CO<sub>2</sub> concentration. Chart speed of the recorder was 60 mm h<sup>-1</sup>, and the full scale was 100 mV. F, flow meter; PD, perma pure drier. (b) Typical time course of change in CO<sub>2</sub> concentration within a chamber of the CC-method.

where a is the time rate of change of the CO<sub>2</sub> concentration in the chamber (slope of line of Fig. 1b) ( $\mu$ l l<sup>-1</sup> min<sup>-1</sup>),  $\rho$  is the density of CO<sub>2</sub> (mg m<sup>-3</sup>), V is the volume of the chamber (m<sup>3</sup>), and S is the basal area of the chamber (m<sup>2</sup>).

## Examination of sampling volume of the air and measuring period

Potential major errors associated with the CC-method are: (i) overestimation of the CO<sub>2</sub> flux due to mass flow of high CO<sub>2</sub> air from soil, resulting from a large sampling volume of air; and (ii) underestimation of the flux due to the increase in the CO<sub>2</sub> concentration within the chamber with time. Thus the effects of the sampling volume and measuring period on the measured value were examined in a laboratory experiment.

As a source of  $CO_2$  emission, approximately 3.0 kg of artificial soil in the container (28 cm  $\times$  35 cm  $\times$  14 cm) was prepared with two replicates.

The artificial soil, which consisted of 3.0 kg d.w. vermiculite, 75 g glucose and 9.0 g mineral nutrients (NH<sub>4</sub>Cl, 7.5 g; NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, 0.75 g; NaH<sub>2</sub> PO<sub>4</sub>, 0.75 g), was inoculated with 60 ml of 10<sup>-2</sup> dilution of soil sampled from cultivated land (volcanic ash soil). Two chambers, 13 cm in diameter and 15 cm in height, were installed 4 cm into the soil for each container. The containers were set in an incubator at 25°C, and the gravimetric soil water content was maintained at 30% by irrigation of deionized water. Three days after the inoculation, the CO<sub>2</sub> emission from the artificial soil was measured using the open flow IRGA method (OFmethod; Nakadai et al. 1993) and the CC-method once a day for a week. The reliability of the CO<sub>2</sub> emission rates measured by the CC-method was examined comparing with those by the OF-method.

To examine the effects of sampling volume on soil respiration rate, the  $CO_2$  emission rates from the artificial soil were compared at sampling volumes of 2.0, 5.0 and 10.0 ml, which correspond to 0.14, 0.34 and 0.68% of the chamber volume, respectively. The air in the chamber was sampled at 10 s, 3 min, 6 min and 9 min after starting the measurement.

In the CC-method, the soil respiration rate is determined by the time rate of change of CO<sub>2</sub> concentration in the chamber (slope of line of Fig. 1b); a long time measurement decreases the slope with time, and underestimates the soil respiration rate. Thus the long time measurement was conducted at three different respiration rates to examine the effect of measuring period on the CO<sub>2</sub> emission rate. When the CO<sub>2</sub> emission rates from the artificial soil were 330, 480, 700 mgCO<sub>2</sub> m<sup>-2</sup> h<sup>-1</sup> the CO<sub>2</sub> emission rate was measured and the CO<sub>2</sub> concentration in the chamber was monitored with 3–10 min intervals for 30–50 min. The time when the slope changed and the CO<sub>2</sub> concentration in the chamber at that time were examined.

#### Field measurements

In order to verify the ability of the CC-method for field measurements, soil respiration rates were measured using the CC- and OF-methods in an experimental field of the National Institute of Agro-Environmental Sciences at Tsukuba Science City, Ibaraki Prefecture in central Japan (36°01′ N, 140°07′ E). The experimental field had been

fallowed since May 1992 and the vegetation was dominated by annual weeds (*Digitaria adscendens* Henry, *Setaria faberi* Herrm. and *Ambrosia artemisiifolia* L. var. *elatior* Desc.), and the soil was volcanic ash. The soil respiration rate from a same plot was measured with the two methods every month from June 1992 to May 1993. The chamber used for the CC-method was 21 cm in diameter and 14 cm in height. The sampling volume of air was 5.0 ml and the time interval was decided from 0.5 min to 5.0 min according to soil respiration rates. The usage of the OF-method was in accordance with Nakadai *et al.* (1993).

#### RESULTS AND DISCUSSION

## Examination of sampling volume of the air and measuring period

If the volume of the sampled air is large, the air pressure in the chamber will decrease, resulting in a mass flow of high CO<sub>2</sub> from the soil and causing the overestimation of soil respiration rate. It is, therefore, important to verify the volume of sampling air for the accurate estimation of the soil respiration. Figure 2 represents the relationship between the relative value of the soil respiration rates by the CC-method to those by the OF-method and the percentage of volume of sampled air to that of the

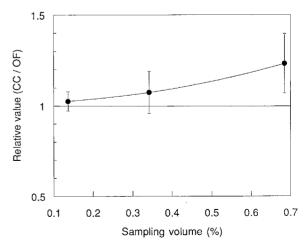


Fig. 2. Relationship between the relative value of soil respiration rates measured by the CC-method to those by the OF-method and the percentage of volume of sampled air to that of the chamber. Each data point denotes the mean of eight measurements  $\pm$  SD.

chamber with three different sampling volumes. When the air volume at each sampling was about 0.2% of the volume in the chamber, the soil respiration rate by the CC-method was nearly equal to that by the OF-method. However, when the sampling volume exceeded 0.6%, the rate by the CC-method was significantly greater than that by the OF-method. Thus, the volume at each sampling should be less than 0.2% of the volume in the chamber.

If the measuring period is long, the CO<sub>2</sub> emission rate will significantly decrease due to the decrease of the CO<sub>2</sub> gradient between air in soil and that in the chamber. Figure 3 represents the changes in the CO<sub>2</sub> concentration within the chamber at three different soil respiration rates. The time rates of change in the CO<sub>2</sub> concentration decreased 20-25 min after the start of the measurements. Namely, the later data points deviated from the straight line with increasing CO2 concentration in the chamber. Moreover, in this case, the critical CO<sub>2</sub> concentrations at which the data points deviate from the straight line ranged from 1000 µl l<sup>-1</sup> to 1500 µl  $1^{-1}$  CO<sub>2</sub> in the chamber. Thus, there are inherent problems, such as the critical time and CO<sub>2</sub> concentration in the chamber, with the measurement for a long time period in this CC-method. Moreover, the problems involved would vary according to soil structure, diffusivity and soil respiration rates. Therefore preliminary measurements are necessary decide the measuring periods and time

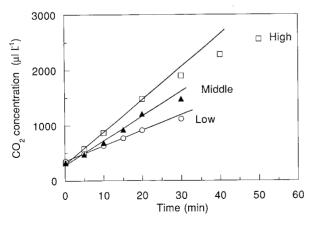


Fig. 3. Time course of changes in the  $CO_2$  concentration within the chamber at different  $CO_2$  emission rates. O, low  $CO_2$  emission rate (330 mg $CO_2$ m $^{-2}$ h $^{-1}$ );  $\blacktriangle$ , middle  $CO_2$  emission rate (480 mg $CO_2$ m $^{-2}$ h $^{-1}$ );  $\Box$ , high  $CO_2$  emission rate (700 mg $CO_2$ m $^{-2}$ h $^{-1}$ ).

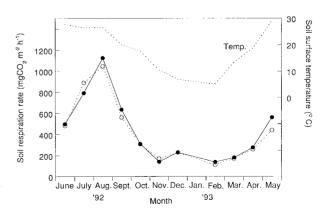
intervals of air sampling. In order to avoid the underestimation of soil respiration, the CO<sub>2</sub> gas within the chamber should be sampled several times over a short time period.

#### Field measurements

Seasonal changes in soil respiration rates obtained by the CC- and OF-methods are represented in Fig. 4. The soil respiration rates increased in July and reached a summer maximum in August, and then decreased during autumn and reached the minimum value of about  $130~\rm mgCO_2~m^{-2}~h^{-1}$  in the following February. Soil respiration rates measured by the CC-method were nearly equal to those by the OF-method through the year. The deviations of the rates by the CC-method were within 20% of those by the OF-method. There were no significant differences in soil respiration rates between the two methods (t-test, P > 0.02).

Freijer and Bouten (1991) reported that in a longer time measurement up to 1 h the increase of  $CO_2$  concentration in a chamber decreases the concentration gradient in the soil, resulting in a decreasing flux. In our results, however, the soil respiration rates by the CC-method were not significantly different from those by the OF-method. This is attributed to the fact that the measurement time of the CC-method was so short (2–15 min) that the  $CO_2$  emission rate would be little affected by the change in the soil  $CO_2$  gradient.

The present results suggest that the CC-method is effective for the measurement of the soil respiration



**Fig. 4.** Seasonal changes in soil respiration rates measured by the CC-method and by the OF-method in the experimental field. ●, CC-method; ○, OF-method; ---, soil surface temperature.

rates and is as reliable as the OF-method. Moreover, the advantages of this closed chamber technique are: (i) it does not greatly disturb the soil environments such as soil temperature, soil moisture and CO<sub>2</sub> concentration, if the measurement is conducted in a short time; (ii) it is not restricted to sites where electricity or special equipments are available; (iii) the chamber is inexpensive to fabricate, transport and use; and (iv) it is possible to obtain many measurement plots in a short time period.

### ACKNOWLEDGEMENTS

We thank Dr Satoh, Mr Usami and Ms Nemoto for their support with this study.

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